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THE EFFECT OF SOLID CARBON RESIDUE FROM TYRE PYROLYSIS ON THE PROPERTIES OF ELASTOMERIC COMPOSITIONS BASED ON BUTADIENE-NITRILE RUBBERS

M.I. Markin, M.E. Solovyov, V.V. Solovyov

Markin Maxim Igorevich, Postgraduate Student; **Solovyov Mikhail Evgenyevich**, Doctor of Physical and Mathematical Sciences, Professor; **Solovyov Vladimir Valentinovich**, Candidate of Technical Sciences, Associate Professor
Yaroslavl State Technical University, 150023, Russia, Yaroslavl, 88 Moskovsky pr.; markinmi@ystu.ru

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Abstract. *The paper presents a comprehensive study of feasibility and effectiveness of using solid carbon residue (pyrolysate), obtained through the low-temperature pyrolysis of worn car tyres, as a functional filler in rubber compound formulations based on butadiene-nitrile rubbers (BNK) of the BNKS-18AMN and BNKS-28AMN grades. The issues of polymer waste disposal and search for cost-effective alternatives to traditional grades of carbon black produced from fossil raw materials provide the relevance of the research. It examines the physicochemical properties of pyrolytic carbon, including its particle size distribution and surface activity. Using vibroreometry (MDR-2000), equilibrium swelling, and physical-mechanical testing, a comparative analysis was conducted of the vulcanization kinetics and properties of vulcanisates containing pyrolysate, compared with reference compounds filled with P 803 carbon black, kaolin, and chalk. Pyrolysate specific surface chemistry and high ash content (including zinc oxide and sulphides) affect significantly on the induction period and the rate of vulcanisation, acting as a filler and as a secondary activator of the cross-linking process. Therefore, the optimisation of dispersion and vulcanising group provide an effective replacement of low-activity fillers and partially replace semi-active carbon black without any significant deterioration in the performance characteristics of the rubbers. The research offers prospects for the development of resource-saving technologies in the manufacture of rubber products.*

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Introduction

The growing amount of waste from polymer materials – and, in particular, worn-out car tyres – is one of the most serious challenges facing modern civilisation. It affects both the environmental and economic aspects of sustainable development. Car tyres are complex, multi-component composite products based on a cross-linked (vulcanised) polymer matrix.

The three-dimensional spatial network formed by strong sulphur bonds (mono-, di- and polysulphide) confers unique elastic and mechanical properties, thermal stability, and chemical resistance on the material. However, these make the tyres extremely resistant to natural biological degradation and environment [1–3].

Under natural conditions, the degradation period for tyre rubber can exceed a century; as a result, landfill sites for such waste become sources of increased fire risk and infectious diseases. Furthermore, the disposal of tyres constitutes a wasteful use of the valuable hydrocarbon resources expended in their production. In this context, recycling minimises environmental damage and serves a source of secondary raw materials – 'urban mining' [4, 5].

Among the existing methods of waste treatment (recycling, mechanical crushing into fines, incineration with energy recovery, and regeneration), pyrolysis – the process of thermal decomposition of organic compounds in an oxygen-free environment – takes a special place. It allows the material to be returned to the chemical cycle by breaking down the polymer chain into low-molecular-weight fractions: pyrolysis gas (a mixture of H_2, CH_4, etc.), a liquid fraction (a mixture of hydrocarbons, analogous to synthetic oil), and a solid carbon residue (char or pyrolytic carbon black – Cb_p) [6, 7]. This solid residue is of greatest interest to the rubber industry. It has the potential to close the carbon cycle by being reintroduced into the production of new rubber compounds.

The tyre pyrolysis process is a complex series of parallel and sequential radical reactions involving decomposition, isomerisation, cyclisation, and carbonisation. The chemical composition and structure of the resulting solid residue depend on the temperature regime and the heating rate [1, 2, 8].

When rubber is heated above 300 °C, thermal dissociation of the S–S and C–S bonds begins. The energy of these bonds (approximately 270 kJ/mol) is lower than that of the C–C bonds in the main chain (approximately 350 kJ/mol). It destroys the vulcanization structure and leads to the degradation of the macromolecules in natural (NK) and synthetic rubbers. There are two main temperature ranges are used for the pyrolysis of worn tyres: low-temperature pyrolysis (450–550 °C) – the rubber matrix undergoes its main decomposition; medium- and high-temperature pyrolysis (>600 °C). The rise of temperature intensifies the processes of secondary cracking and gasification of carbonaceous deposits. This results in the cleaning of the pore surfaces and increasing in the specific surface area of the solid residue. However, graphitisation of the structure may occur, reducing the surface activity of the material [2, 6].

The solid carbon residue from rubber pyrolysis (CB_p) differs fundamentally from conventional industrial carbon black. Industrial carbon black is synthesised 'from zero' from gaseous or liquid feedstocks via incomplete combustion (the 'bottom-up' method). CB_p is a composite consisting of primary carbon black aggregates, initially incorporated into tyre rubbers, modified by carbonaceous rubber decomposition products and enriched with inorganic components (ash) [4, 7, 9].

The ash content makes up 10–20% of the mass of CB_p, with a key role in its performance as a filler. The main components of the ash are zinc oxide (ZnO) and zinc sulphide (ZnS). They are formed as a result of the transformation of vulcanisation accelerators, as well as silicon dioxide (SiO_2), and calcium compounds. The presence of these chemically active compounds on the surface of carbon particles forms a specific 'surface chemistry' capable of influencing the

kinetics of sulphur vulcanisation, accelerating or slowing down the cross-linking reactions depending on the conditions [10, 11].

Among the various types of synthetic rubbers, a distinct group is formed by butadiene-acrylonitrile rubbers (BNR, NBR), which are copolymers of butadiene and acrylonitrile (AN). The presence of polar nitrile groups (-C≡N) in the macromolecule determines the unique set of properties possessed by BNC vulcanisates: high resistance to aliphatic hydrocarbons (oils, fuels), enhanced adhesion to various substrates, and good physical and mechanical properties [12, 13].

The low activity of CB_p is tested in rubber compounds for rubber goods. Its formulations include low-activity fillers, typically added in large quantities. The most common elastomeric compounds used in the manufacture of rubber goods are those based on butadiene-nitrile rubbers.

The specific nature of the interaction between fillers and the polymer matrix is determined by the polarity of the matrix. Non-polar rubbers (NK, SKI, SKD, BSK) interaction with the carbon filler is determined primarily by Van der Waals dispersion forces and physical chain entanglement. However, BNK have dipole-dipole interactions and the possibility of hydrogen bonding between functional groups on the filler surface and polar groups of the rubber [12, 13].

When using solid pyrolysis products in a BNC, two competing factors should be considered:

1. Affinity for carbon surfaces: The carbon nature of CB_p ensures compatibility with the hydrophobic regions of the polymer chain (butadiene units).

2. Effect of ash components: Polar inorganic inclusions (ZnO, SiO_2, $CaCO_3$) in the pyrolysate may interact specifically with nitrile groups, altering the mixture's rheology and the kinetics of matrix formation.

Formulation of the research objective

Despite the considerable amount of research into tyre pyrolysis, the practical application of the solid residue in critical rubber compounds remains limited. The main limitations are the instability of the CB_p composition, high ash content, and the presence of incomplete decomposition products (resins) on the surface. They block active sites and prevent effective interaction with the rubber.

The existing literature data indicate that CB_p has a contradictory effect on rubber properties: in some cases, a reinforcing effect is observed, similar to that of semi-active carbon black grades; in others there is a sharp decline in strength and elasticity. The effect of pyrolysis products on the vulcanisation kinetics of blends based on polar rubbers, such as BNK. It remains a particularly understudied area as the presence of residual sulphur and zinc oxide in the filler can significantly distort standard cross-linking mechanisms.

The purpose of this study is to systematically investigate the kinetic patterns of vulcanisation and the evolution of the physical and mechanical properties of rubber compounds based on tyre pyrolysis residue (TPR) when traditional fillers (P 803 carbon black, kaolin, and chalk) are partially or completely replaced by solid carbon residue from tyre pyrolysis. Special

attention is paid to analysing the correlations between the filler composition, the vulcanisation parameters, and the macroscopic properties of the material.

Objects and methods of research

Characteristics of the raw materials

Industrial grades of Russian-made butadiene-nitrile rubbers are widely used in the rubber industry for the manufacture of oil- and petrol-resistant products. They were selected as the polymer matrix (binder) [12–14]:

1. BNKS-18AMN: Synthetic butadiene-acrylonitrile rubber with an acrylonitrile content of 17–20%. It is characterised by high frost resistance (glass transition temperature of approximately –50 to –55 °C) and satisfactory oil resistance. The 'M' index indicates softness (low Mooney viscosity); 'H' indicates the use of a non-darkening antioxidant 8.

2. BNKS-28AMN: Rubber with a natural rubber content of 26–30%. It offers a balanced combination of oil resistance, heat resistance, and elasticity. It is used in the production of general and specialised rubber goods (cuffs, gaskets, sleeves) [15].

Traditional materials were used as reference fillers to compare the effectiveness of the pyrolysate:

• **Technical carbon P 803 (equivalent to ASTM N880/N990)** [16]: A low-activity furnace-grade technical carbon obtained by the thermal oxidation of liquid hydrocarbon feedstock. It is characterised by a low specific surface area (12–18 m^2/g) and a low structural index (oil absorption of 60–75 $cm^3/100$ g). It ensures high bulk density and improves the processing properties of the mixtures.

• **Kaolin ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$):** A natural aluminosilicate and inert, light-coloured filler reduces the cost of mixtures and increases stiffness.

• **Natural chalk ($CaCO_3$):** An inert filler added to reduce costs and control rheological properties.

Test material (subject of the study): Solid carbon residue from tyre pyrolysis (hereinafter – pyrolysate or CB_p). The material was obtained by low-temperature pyrolysis (450–500 °C) of a mixture of worn car tyres in an experimental rotating horizontal cylindrical reactor. In [17], the physicochemical characteristics of the liquid pyrolysis fraction obtained in this reactor were presented. This study investigates the potential for using the solid carbon fraction. Before being added to the rubber compounds, the product underwent preliminary processing: removal of the steel cord, coarse crushing, and subsequent fine grinding in an impact disintegrator to improve dispersion. Table 1 shows the particle size distribution of the pyrolysate, determined by sedimentation turbidimetry using an FSH-4 automatic photosedimentometer (Russia). The average particle size was 6.7 μm.

Table 1. Dispersion of ground pyrolysate

Equivalent sphere diameter	Mass fraction of particles
Less than 20 μm	87
Less than 10 μm	52
Less than 5 μm	19
Less than 1 μm	1

The chemical composition of the pyrolysate is characterised by a high ash content (12–15%, predominantly ZnO, SiO_2, Ca and S), the presence of a carbonaceous matrix (80–85%) and residual sulphur (1.5–2.5%) [1, 2, 6].

Methodology for the preparation of rubber compounds

The rubber compounds were prepared in the laboratory using an Lb 320 160/160 mixer, in accordance with standard mixing procedures for butadiene-nitrile rubbers. The temperature of the rollers was maintained within the range of 30–50 °C to prevent premature vulcanisation (scorching), given the high activity of the natural rubber during mechanical processing [13, 14].

The order of adding the ingredients (mixing mode) was as follows:

1. Rubber plasticisation (formation of a continuous skin on the front roller).

2. Addition of activators (zinc oxide, stearic acid) and dispersants.

3. Additives (carbon black, pyrolysis residue, kaolin, chalk) should be introduced in small batches to ensure even distribution and wetting of the particle surfaces by the rubber.

4. Addition of plasticisers.

5. Addition of the vulcanising group (sulphur, accelerators: Altaks, Kaptaks, DFG, thiuram, etc.) during the final stage of mixing.

After mixing, the rubber compounds were sheeted and left to cure for 24 hours to allow internal stresses to relax before testing.

Methods for studying vulcanisation kinetics

A range of physicochemical and mechanical analytical methods was used to comprehensively assess the effect of pyrolysate on the properties of elastomeric composites [10, 14, 18].

Key method for study the kinetics of vulcanisation was vibroreometry [19] on a rotorless rheometer MDR-2000 (Moving Die Rheometer). The principle of operation is based on measuring the torque required to maintain harmonic vibrations of the lower half-form containing the sample at a specified temperature and frequency. However, the upper half-form remains stationary and is fitted with a torque sensor.

During the test, rheometric curves (the relationship between torque S' and time t) were recorded. It allows us to determine the following critical process parameters:

• M_L (minimum torque, N·m). Indicates the viscosity of the rubber compound at the test temperature prior to the onset of vulcanisation. This indirectly indicates the extent of physical interaction between the rubber and the filler, as well as the processability of the mixture.

• M_H (maximum torque, N·m). It is proportional to the shear modulus of a fully cross-linked vulcanised product. It serves as a measure of the density of cross-links and the stiffness of the material.

• $\Delta M = M_H - M_L$. A difference in torque that correlates with the chemical density of the vulcanisation matrix.

• t_{s1}/t_{s2} (start time of sub-vulcanisation). The time taken for the torque to increase by 1 or 2 units relative to M_L. It determines the induction period of the reaction and the workability of the mixture (the time until premature setting begins).

• t_{90} (optimal vulcanisation time). The time to reach 90% of maximum vulcanisation ($M_{90} = M_L + 0.9(M_H - M_L)$). This is the key technical parameter for determining the vulcanisation conditions for the products.

• Vulcanisation rate (R_V). It is defined as the tangent of tangent line angle inclination to the rheometric curve on a linear section of the main curing period (Cure Rate Index, CRI).

The kinetic analysis was performed in an isothermal regime at temperatures of 143 °C, 150 °C, 160 °C and 170 °C. It enabled the activation energy of the vulcanisation process (E_a) to be calculated using the Arrhenius equation.

To assess the structural parameters of the vulcanised network (cross-linking density), the equilibrium swelling method was used in a thermodynamically favourable solvent (toluene). The theoretical basis of the method is the Flory-Renner equation. It relates the degree of polymer swelling to the density of the network nodes [10, 14, 18].

The following parameters were determined:

• Mass fraction of the sol fraction (S). The proportion of uncross-linked polymer that can be extracted with a solvent.

• Equilibrium swelling degree (Q_∞). The maximum amount of solvent absorbed by the sample.

• The volume fraction of rubber in the swollen gel (V_r).

• The average molecular weight of the chain segment between the network nodes (M_c), calculated using the formula:

$$M_c = \frac{-\rho_p V_s \left(V_r^{1/3} - \frac{V_r}{2}\right)}{ln(1 - V_r) + V_r + \chi V_r^2} \quad (1)$$

where ρ_p is polymer density, V_s is molar volume of the solvent, χ is Huggins 'polymer-solvent' interaction parameter.

This method makes it possible to distinguish between the contributions of physical interactions (interactions, adsorption on the support) and chemical cross-links to the overall structure of the material.

Methods for investigating the physical and mechanical properties of rubbers

This paper presents a standard set of tests used to evaluate the elastic-deformation, strength, and special properties of technical rubbers based on butadiene-nitrile rubbers [20, 21]. Table 1 presents the methods used to investigate the physical and mechanical properties of rubbers, their main characteristics, and application. The rubber compounds, vulcanised at an optimum temperature of 151 °C using standard methods [22–24], were subjected to testing.

Study results

The experimental programme involved a systematic assessment of the effectiveness of replacing traditional P 803 carbon black fillers with solid carbon residue (pyrolysate) in various BNK-based formulations.

The first series of experiments investigated a base formulation based on BNKS-18 AMN rubber, designed for critical rubber products operating under fairly hard conditions. The control mixture contained 103 parts by weight of P 803 technical carbon per 100 parts by

weight of rubber. The effect of partially replacing the P 803 technical carbon with pyrolysate was evaluated.

Upon the addition of the pyrolysate, the following trends were observed in the changes to the rheometric characteristics.

As the proportion of pyrolysate increased, a slight change in the mixture's viscosity was observed. At a 50% replacement level, an increase in the ΔM index and the maximum vulcanisation rate (R_h) was defined. This indicates an improvement in cross-linking efficiency. The reason for this effect is the chemical composition of the ash fraction of the pyrolysate. The ash contains a significant amount of zinc oxide (ZnO) and residual sulphur migrating into the rubber matrix and acting as additional activators and cross-linking agents. It enhances the action of the main vulcanising group [1, 10, 11]. At the same time, a reduction in the onset time of sub-vulcanisation was observed. This confirms the hypothesis regarding the catalytic activity of impurities in the pyrolysate. The presence of unreacted residues of accelerators and active sites on the surface of CB_p reduces the activation energy for the onset of the sulphur addition reaction. It is a critical factor for the processing technology. It increases the risk of scorching (premature vulcanisation). According to swelling degree studies, rubbers with the solid carbon residue from tyre pyrolysis (a secondary product content of approximately 30 wt.% or ~25% of the total filler content) are characterised by a degree of chemical cross-linking at the reference level.

The results of physical and mechanical tests on vulcanised compounds (Table 2) show that pyrolysate is not an equivalent substitute for P 803 carbon black. It is partially substituted; a decrease in the stress at 100% elongation; the tensile strength is observed. At the same time, other parameters, such as relative elongation at break, tear resistance, hardness and rebound elasticity, change insignificantly according to statistical analysis. Therefore, they meet the standards for stress and strength. Moreover, the introduction of pyrolysate as a partial substitute for P 803 provides the retention of the property profile by utilising the synergistic effect of the ash components with the main vulcanising system.

Table 2. Physical and mechanical properties of rubber compounds based on BNKS-18 AMN rubber

Indicator	Average value and coefficient of variation	Ratio of technical carbon P 803 to carbon residue, % by mass		
		120 : 0	112.5 : 7.5	105 : 15
Relative stress at 100% elongation f_{100}	Average, MPa	7.56	6.99	4.16
	$K_в$, %	3.04	4.01	3.04
Relative tensile strength f_p	Average, MPa	10.12	8.64	7.17
	$K_в$, %	2.01	2.10	2.01
Relative elongation at break ε_p	Average, %	162	140	218
	$K_в$, %	8.05	5.05	8.05
Tensile strength B	Average, kN/m	19.5	15.2	23.1
	$K_в$, %	14.5	3.5	15.1
Shore hardness	Average, rel. units	72.4	74.1	70.9
	$K_в$, %	1.3	1.4	3.7
Rebound elasticity	Average, %	34.5	34.8	31.3
	$K_в$, %	7.9	7.8	6.6

In the next series of experiments, the possibility of partially replacing P 803 technocarbon in the mixture with the more polar BNKS-28AMN rubber was investigated. The following data were obtained (Table 3).

Table 3. Physical and mechanical properties of rubber compounds based on BNKS-28 AMN rubber

Indicator	Average value and coefficient of variation	Ratio of technical carbon P 803 to carbon residue, % by mass		
		103 : 0	97 : 6	90 : 13
Relative stress at 100% elongation f_{100}	Average, MPa	0.96	1.17	1.21
	K_a, %	4.5	3.4	2.6
Relative tensile strength f_p	Average, MPa	4.96	4.92	4.72
	K_a, %	3.3	7.3	3.9
Relative elongation at break ε_p	Average, %	504	473	441
	K_a, %	4.7	5.2	5.4
Relative residual elongation	Average, kN/m	20.6	10.7	11.8
	K_a, %	2.2	4.5	2.1

Therefore, use of pyrolysate in rubbers based on more polar rubber is more effective. An increase in the tensile strength at 100% elongation and a decrease in the residual elongation are observed. The relative elongation at break decreases slightly and agrees with the standard limits. According to statistical analysis, parameters have changed insignificantly. Furthermore, compounds under study contain carbon black, chalk, and kaolin. These can be partially replaced without any significant change in the physical and mechanical properties.

Vulcanised compounds with the chalk replaced by pyrolysate demonstrated higher hardness and elastic modulus values maintaining an acceptable level of strength. Pyrolysate can effectively replace mineral fillers, acting as a semi-reinforcing agent rather than merely an inert diluent. However, to implement such a substitution in practice, a significant adjustment to the vulcanising system (reducing the content of sulphur and accelerators) is required to prevent under-vulcanisation.

Discussion of study results

The experimental data obtained clearly indicate that the solid product of tyre pyrolysis is not an inert component. Its effect on the kinetics of vulcanisation can be described by a 'dual-action' mechanism:

1. During pyrolysis, part of the sulphur bound in the sulphide bridges of tyre rubber is not removed as H_2S or mercaptans. It remains in the structure of the solid residue as inorganic sulphides (ZnS) or sulphur chemically bound to carbon. Under re-vulcanisation conditions at temperatures of 150–160 °C, sulphur can undergo exchange reactions and participate in the formation of new cross-links.

2. A high ZnO content (up to 4–5% by mass of the pyrolysate) plays a critical role. In the classical mechanism of sulphur vulcanisation, zinc oxide reacts with stearic acid to form zinc stearate. Otherwise, they then form active complexes with the accelerator and sulphur. The introduction of pyrolysate effectively increases the concentration of the activator in the system. Furthermore, as the ZnO in the pyrolysate is distributed at the molecular level or in the form of

nanoparticles within the carbon matrix. Its reactivity may be higher than that of crystalline ZnO introduced separately.

These factors explain the observed reduction in the t_s1 time and the increase in the vulcanisation rate. To describe the kinetics of the process, autocatalytic models of the Kamal-Surur type are used. The rate constants depend on the concentration of the pyrolysate as a source of catalytic sites.

The reduction in strength properties when replacing P803 carbon black with pyrolysate is due to several physicochemical factors:

1. Sedimentation analysis and electron microscopy data show that pyrolysate particles have a wide range of sizes, including fractions larger than 10–40 μm. In rubber, such large agglomerates act not as reinforcing fillers but as stress concentrators (structural defects), initiating crack growth during deformation. For effective reinforcement, the particle size must be comparable to that of carbon black (<100 nm for aggregates).

2. The surface of pyrolytic carbon is often covered by a layer of amorphous carbon (coke) formed during the decomposition of the oil and additives. This layer blocks the active functional groups (hydroxyl, carbonyl) for interaction with the polar nitrile groups of the BNK. The absence of strong interphase interaction leads to delamination of the matrix from the filler under load (de-vetting effect), which reduces strength.

3. High surface polarity, caused by ash (SiO_2, ZnO), may prevent the carbon part of the matrix from being wetted by the non-polar segments of the rubber. Although, for BNK this effect is less significant due to the polymer's inherent polarity.

Based on the patterns identified, the following steps are required to transform the pyrolysate from a cheap filler (diluent) into a functional component:

- Use of jet mills or wet grinding to break down agglomerates to the submicron level.
- Acid treatment (HCl, H_2SO_4) to remove excess ash. This will expose the carbon surface, increase the specific surface area (BET), and raise the content of active oxygen groups
- When using raw pyrolysate, the dosage of the main vulcanising group (sulphur and accelerators) should be reduced in terms of the pyrolysate's 'internal reserve'; vulcanisation retarders (PVI) should be introduced to control the induction period.

Conclusion

The study conducted concludes the following:

Pyrolysate can be used as an effective substitute for low-activity mineral fillers (chalk, kaolin) and as a partial substitute for semi-active carbon black (P 803) in dosages of up to 20% by mass without any significant loss of the performance properties of oil- and petrol-resistant rubbers. The use of pyrolysate is more effective in rubbers based on nitrile-rich rubbers.

The introduction of pyrolysate significantly alters the vulcanisation kinetics, acting as a secondary activator. The results in a reduction in the induction period and an increase in the cross-linking rate necessitates the adjustment of the vulcanising group composition (reducing the content of accelerators, introducing sub-vulcanisation inhibitors).

At low loading levels, the pyrolysate promotes the formation of a dense vulcanisation network due to the participation of its own active components (S, ZnO).

For industrial implementation, preliminary preparation of the pyrolysate is required. It includes fine grinding to remove fractions that act as stress concentrators.

Overall, the use of solid pyrolysis products in natural rubber-based compounds is a promising area. It enables the implementation of circular economy principles, reduces production costs, and addresses the issue of tyre waste disposal.

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